Analysis of Perchlorate- Right or Wrong?

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What are the Issues?

■ Perchlorate is a serious recognized problem in California and Nevada

■ There are known sources for perchlorate

■ Is the analytical method capable of meeting the needs?

■ Is perchlorate a serious problem across the country?

■ Is it localized in occurrence or like VOCs-ubiquitous?

First Cut Approach

- Urgent Problem in California

  - strong interest in monitoring to evaluate concern

- California Department of Health Services (CDHS) developed a method for trace analysis of perchlorate

- Optimization of the CDHS Method for rapid throughput and high sensitivity

Optimization of the Method

- Ion Chromatography

  - Selection of Column - AS5

- Avoiding False Directions

  - Chloosing the right eluant, regenerant, and conditions

- MDL Determination and P&A

- Typical Chromatograms

- Potential Interferences - avoiding them

CLO$_4$ Analysis - doing the numbers

- ■ MDL = 0.8 ppb
  - (based on 4 ppb spike)

- ■ MRL = 4 ppb

- ■ Linear Range = 2 ppb to >100 ppb

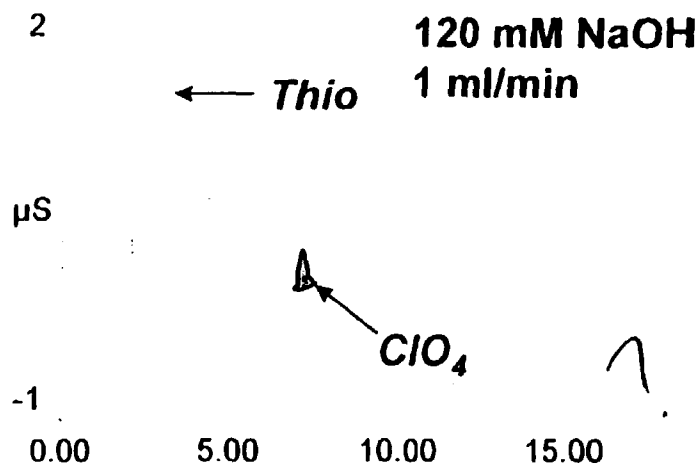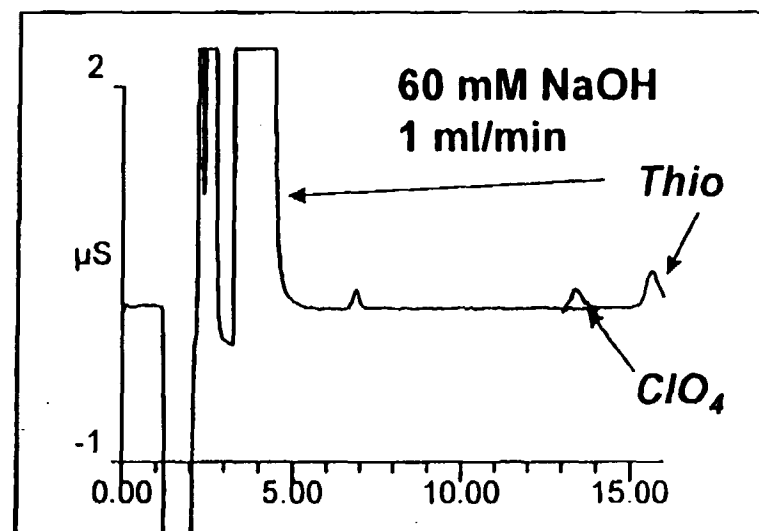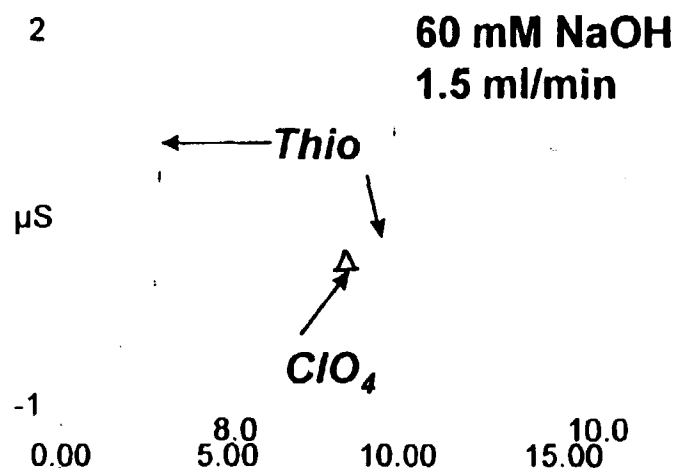- ■ Recovery of check standards at the CA Action Level (18 ppb) = 101-106%

Instrument Operating Conditions

- Dionex Model DX500

- AG5 Guard + AS5 Ionpac Column

- 750 ul Injector Loop

- Eluent - 120 mM NaOH + 2mM
  p-cyanophenol

- Regenerant - AMMS-II with 60mN H_2SO_4

  - Note - this is important for low noise

- Conductivity background <15 uS

Eliminating Interferences-thio

**60 mM NaOH
1.5 ml/min**

Thio

μS

ClO₄

2

-1

8.0   10.0
0.00   5.00   10.00   15.00

**60 mM NaOH
1 ml/min**

Thio

μS

ClO₄

2

-1

0.00   5.00   10.00   15.00

**120 mM NaOH
1 ml/min**

Thio

μS

ClO₄
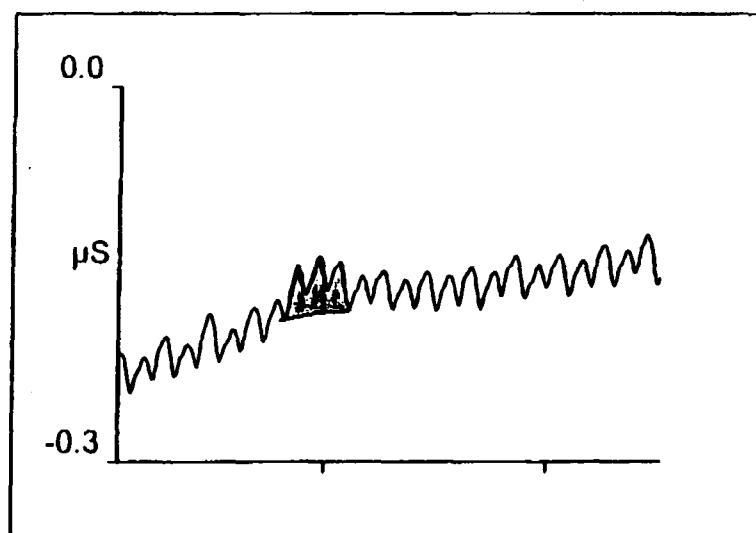
2

-1

0.00   5.00   10.00   15.00

This ▮ows the effect of changing eluant and flow rate based on attempting to use the SRS supressor, which can not handle 120 mM NaOH
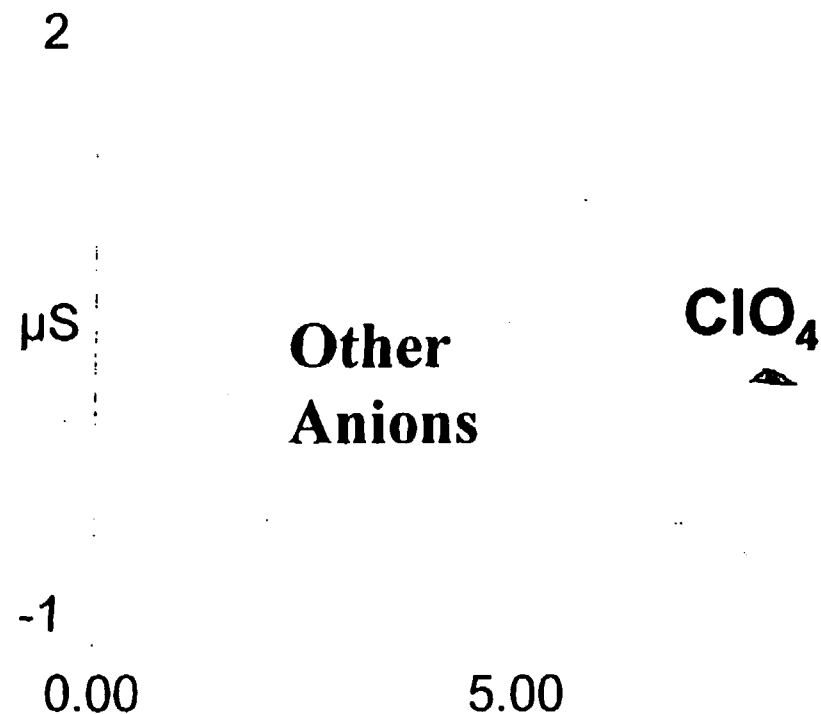
Chromatograms of Standards
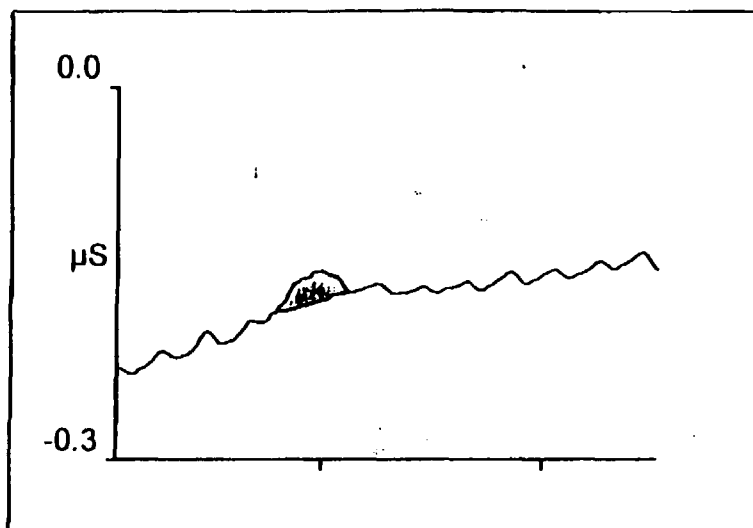
4 ppb CLO_4
- smoothed

4 ppb CLO_4
- no smoothing

Note: Peak with a new column is much sharper than these examples

Typical Full Scale Chromatogram
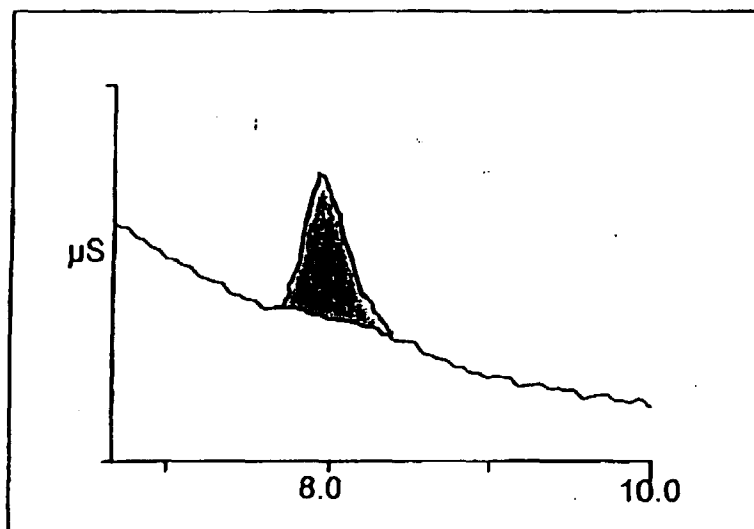
2

μS

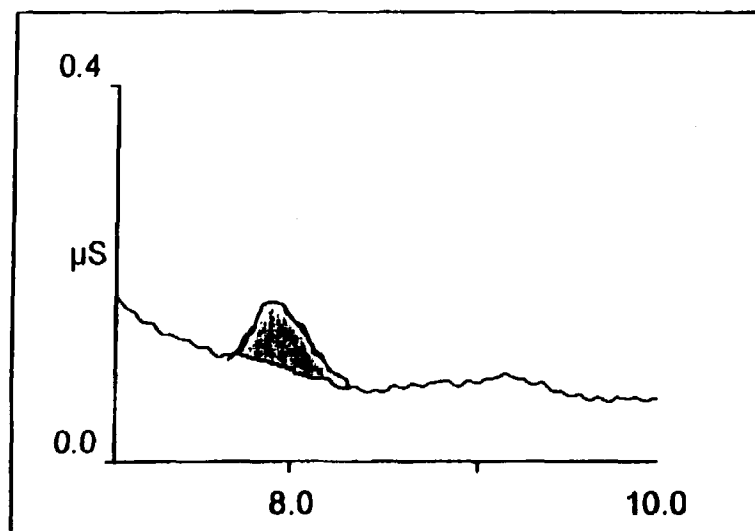Other
Anions

ClO_4

-1

0.00   5.00

Sample w 7 ppb ClO_4

Chromatograms of Samples

Sample w 30 ppb ClO_4
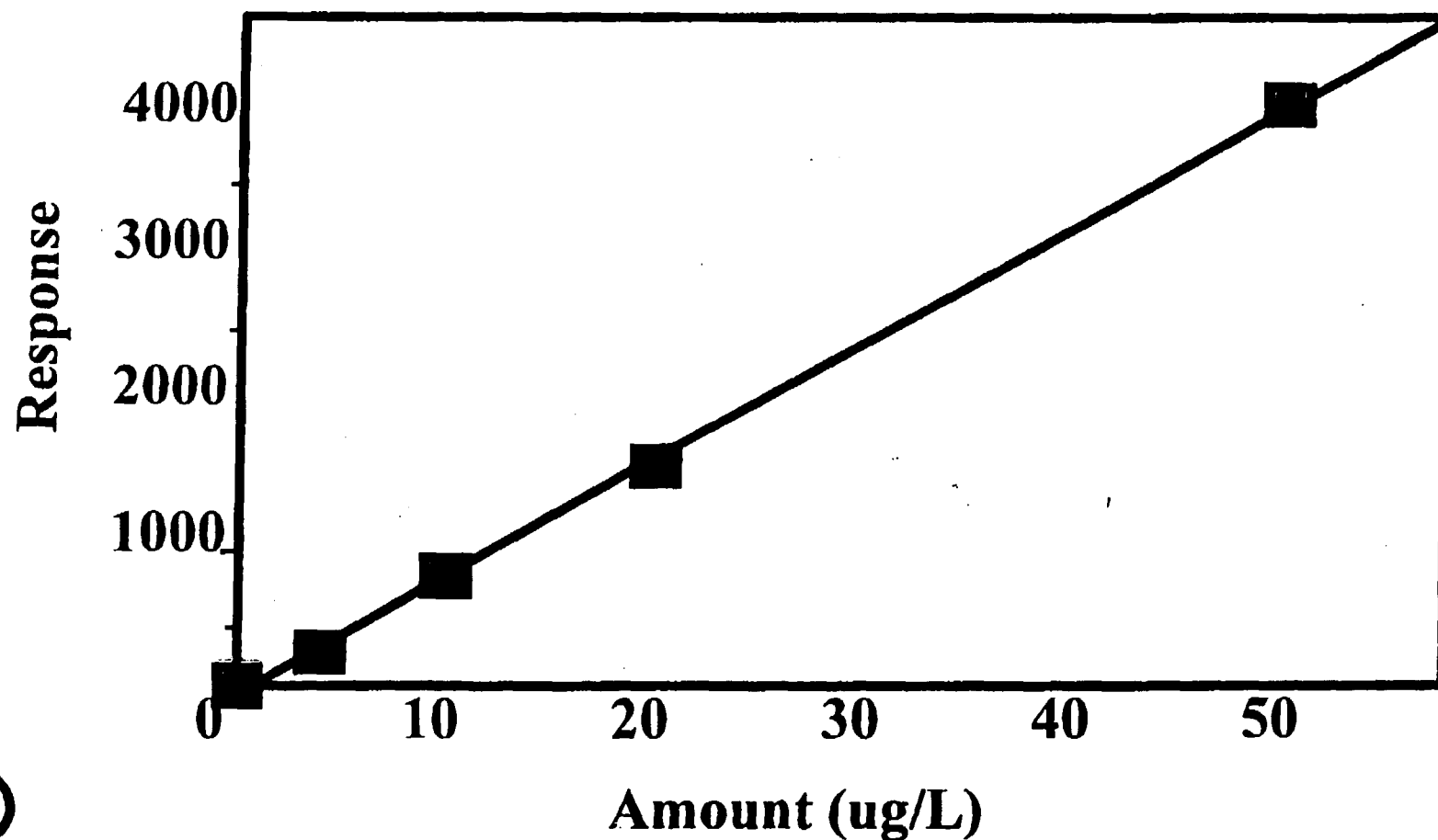
Sample w 7 ppb ClO_4 no smoothing

Perchlorate Calibration Curve

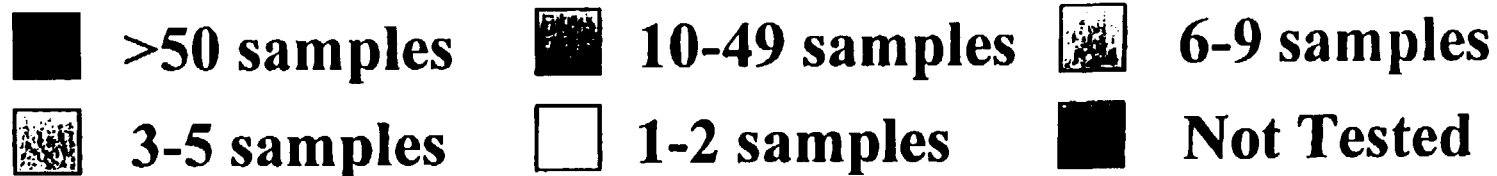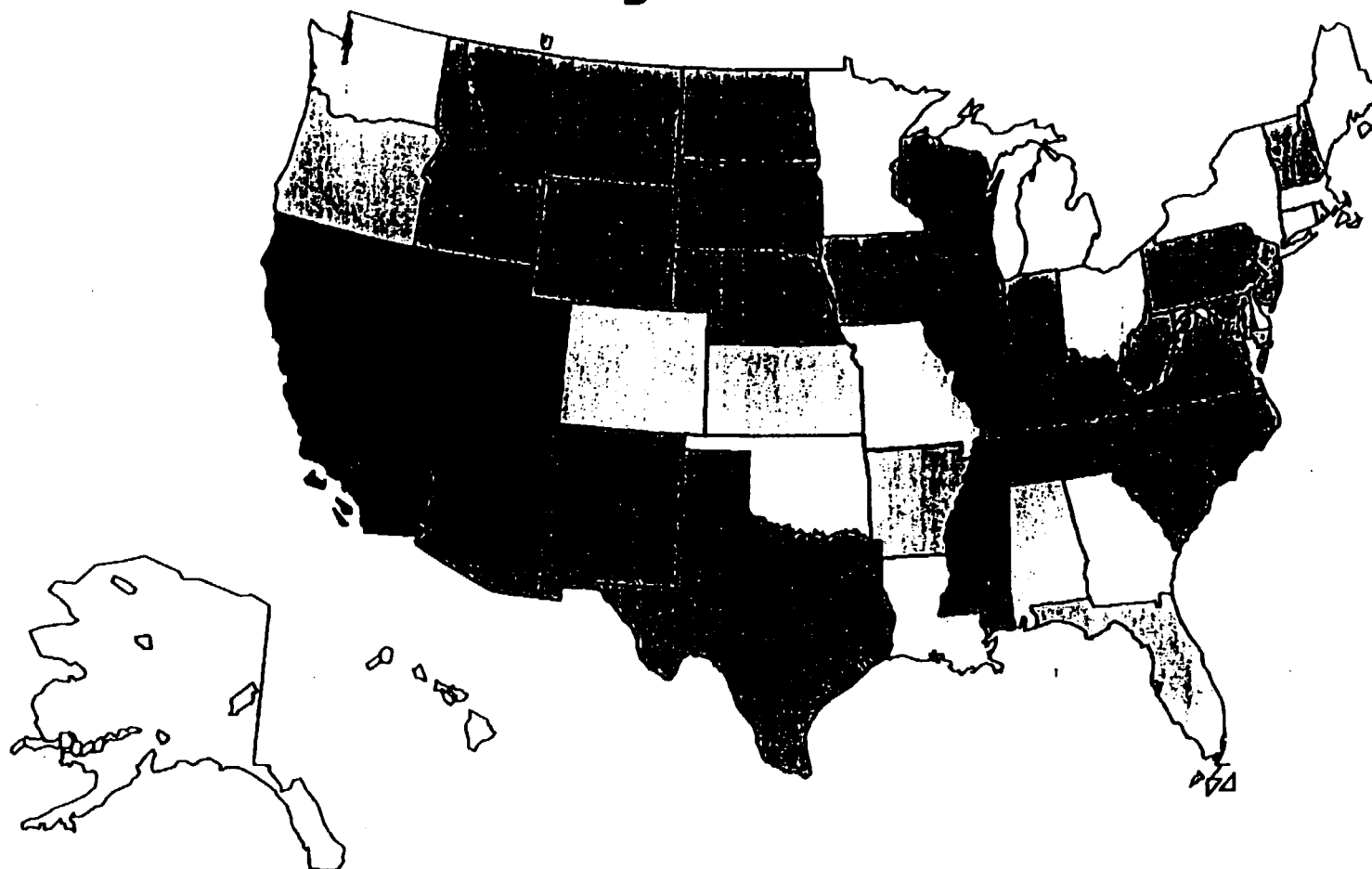$$Amt = 0.0126 * Resp + 0.8056 \quad r^2 = 0.999$$

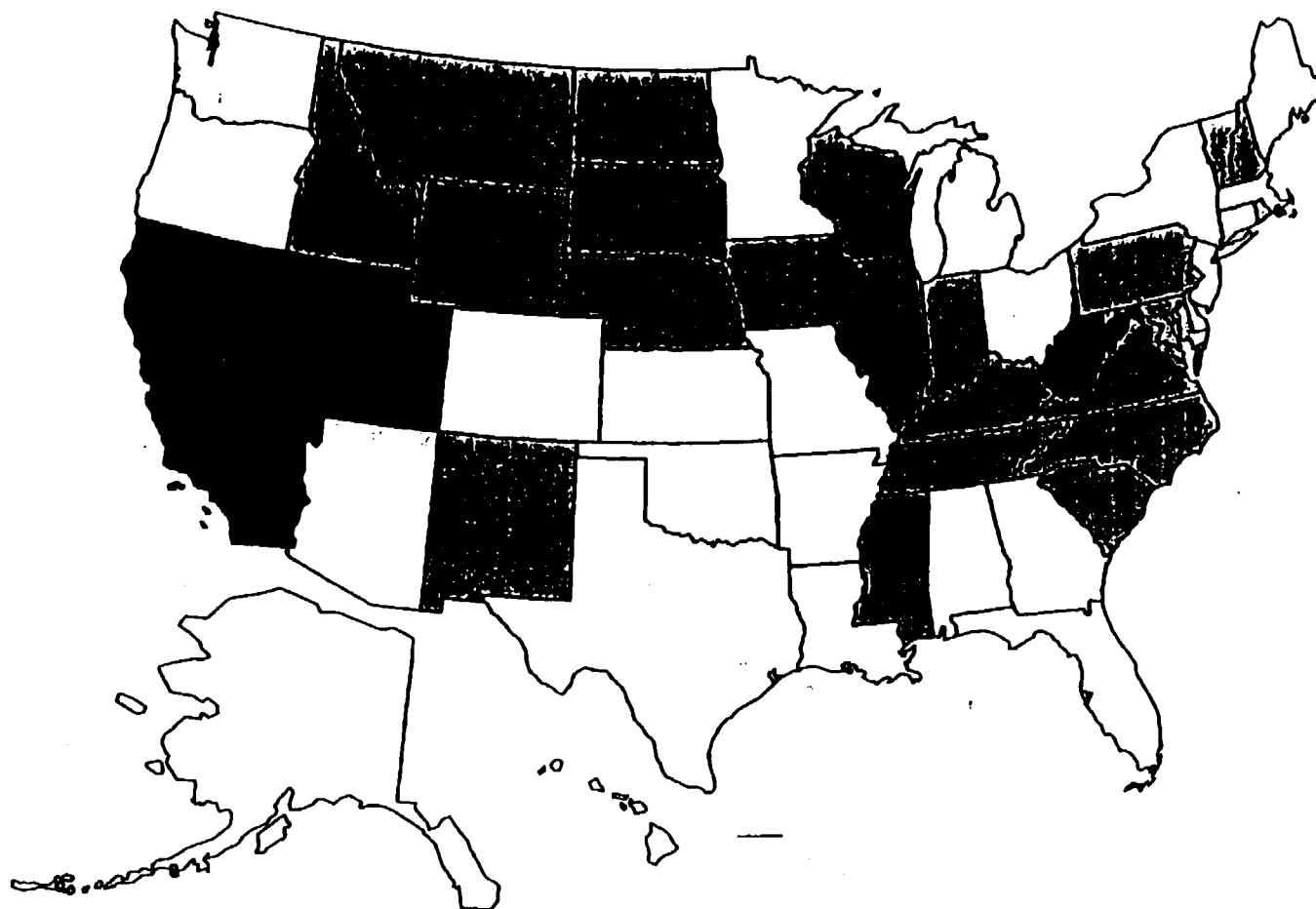# Results - National Overview

- 700+ Samples tested in 2 months

    - Mostly California and Nevada

- 25 States represented

- Both ground and surface waters tested

Number of Samples Analyzed by State

■ >50 samples		▓ 10-49 samples		▒ 6-9 samples	
▒ 3-5 samples		□ 1-2 samples		■ Not Tested	

Distribution of CIO$_4$ Hits by State
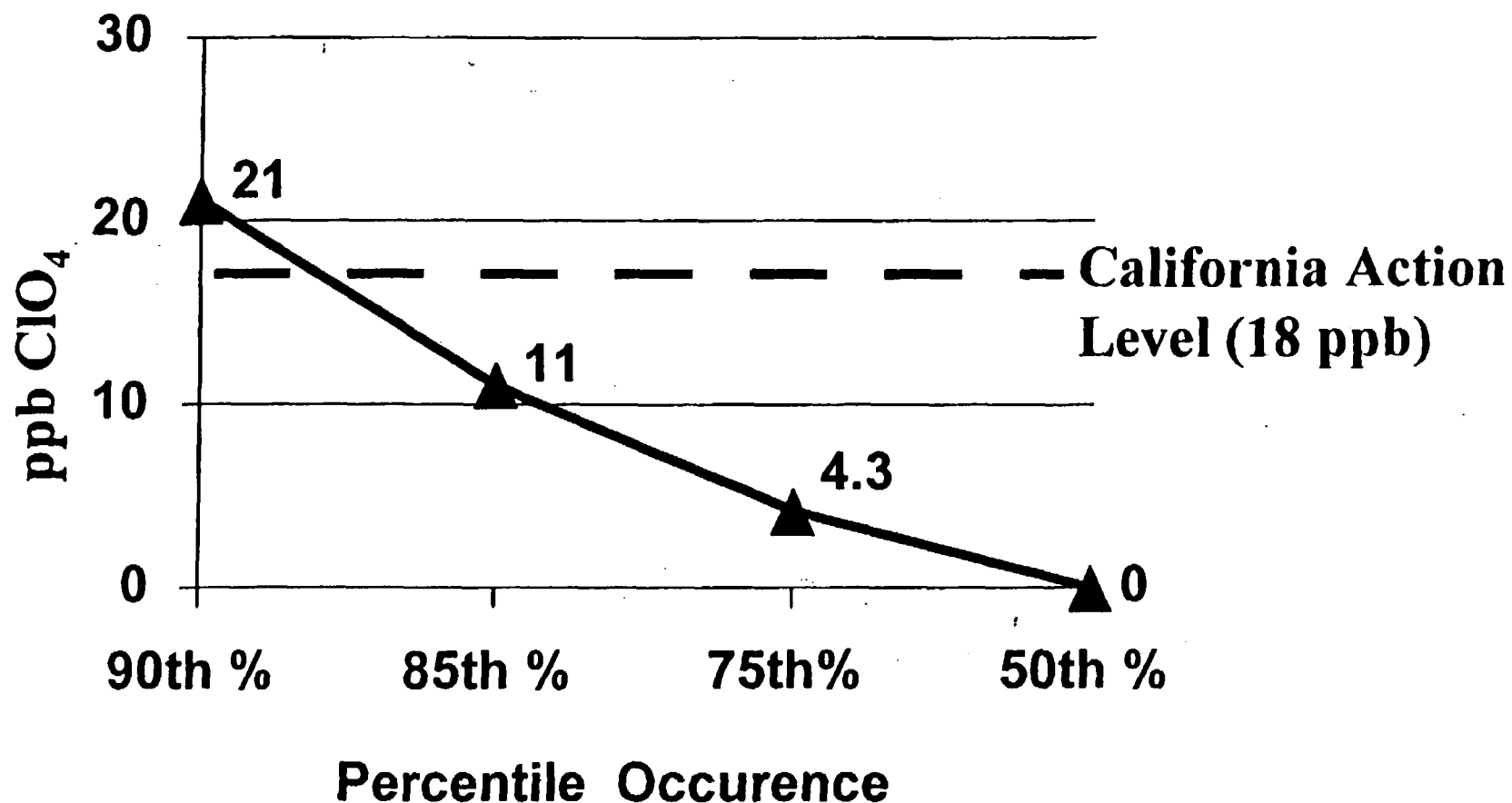

■ Hits □ No Hits ■ Not Tested

National Overview - Summary

- California - >20% Hits

- Nevada - >60% Hits (targeted samples)

- Utah - >50% Hits (targeted samples)

- West Virginia - 10% Hits

- Others - 0% Hits

- SOCs - nationally <1% Hits

National Overview-700+ samples

Conclusions

■ Determination of perchlorate at trace levels is a non-trivial analytical task

■ Perchlorate is NOT a national problem per se as far as levels and frequency

■ Perchlorate is much more common than SOCs or VOCs, which are regulated nationally

Conclusions - continued

- The places where it does occur at <u>high</u> concentrations are "predictable" based on historic or current use

- When it does occur, it is widespread in a given area because it is so mobile

- There will likely be other "perchlorates" in the future

- Treatability is the ultimate problem